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Title: Bulk Nanolayered Composites: Interfacial Influence on Microstructural Evolution at Large Plastic Strains

Author(s): Mara, Nathan A.  
Carpenter, John S.  
Han, Weizhong  
Zheng, Shijian  
McCabe, Rodney J.  
Wang, Jian  
Beyerlein, Irene J.

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# **Bulk nanolayered composites: Interfacial influence on microstructural evolution at large plastic strains**

**N.A. Mara**

Center for Integrated Nanotechnologies

Los Alamos National Laboratory

Co-Authors:

J. Carpenter, W.Z. Han, S. Zheng, R. McCabe, J. Wang, I.J. Beyerlein



# Layered composites: Improving mechanical properties since Egyptian times

Plywood—found in Egyptian tombs dating to 3500 BC



--courtesy Amazon.com



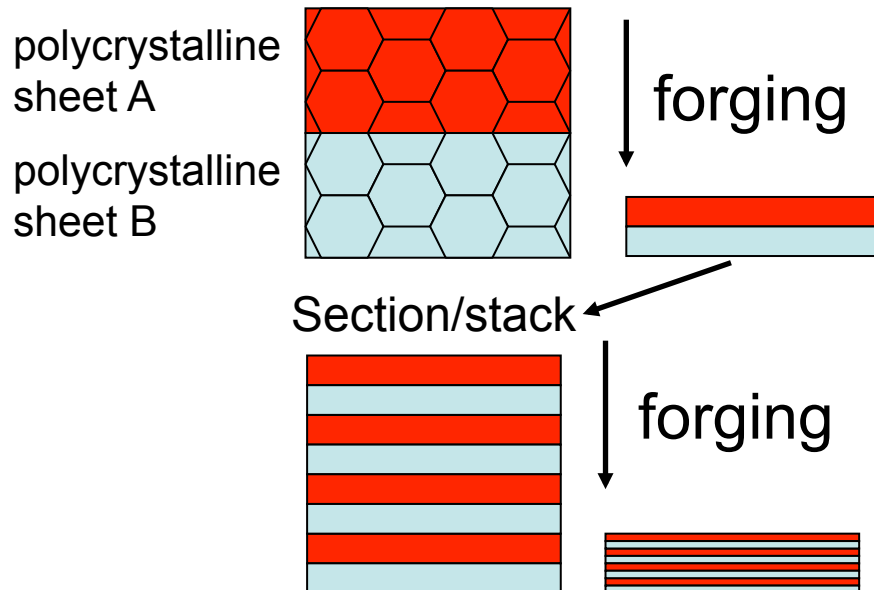
Damascus steel production began ~300BC

Moro barung from Philippines—courtesy Wikipedia

## Metallic lamellar composites: Damascus steel

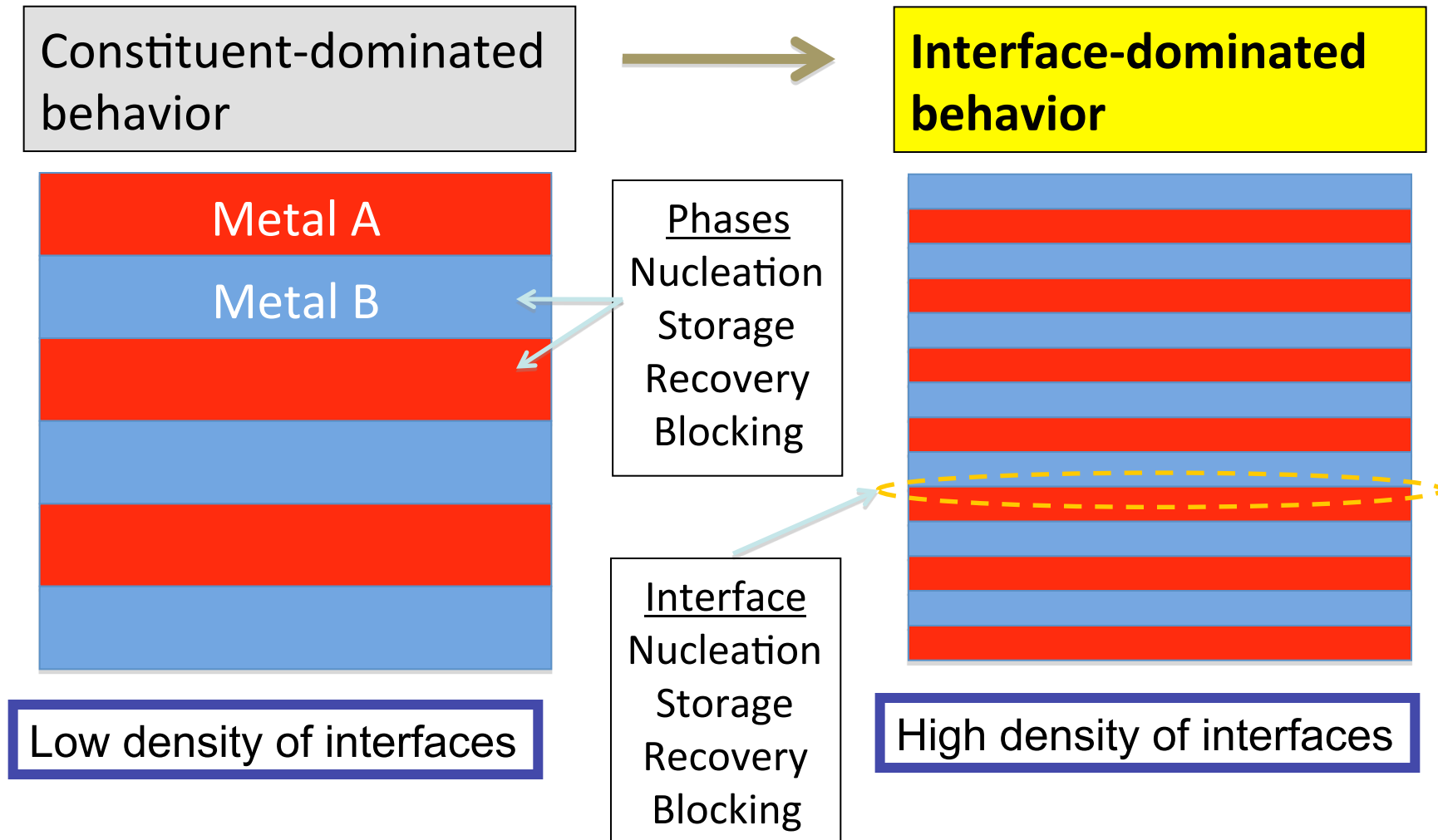


Two different types of steel repeatedly forge-welded together—high strength and toughness combined



Moro barung from Philippines—courtesy Wikipedia

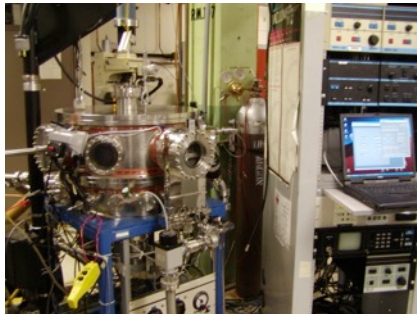
# Defects and Interfaces: Interface-driven material behavior



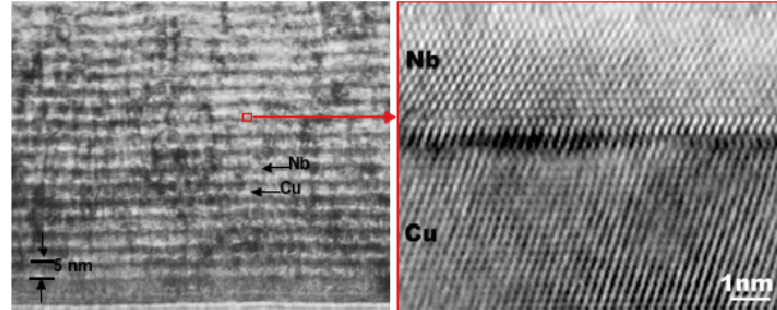


# Extraordinary behavior of Physical Vapor Deposited (PVD) multilayers

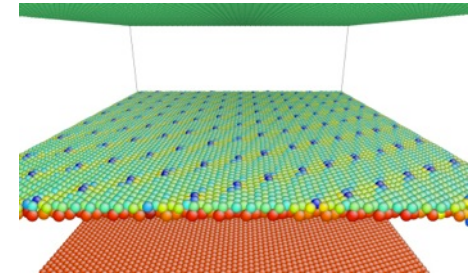
PVD multilayer fab



Cu-Nb 2.5 nm layers



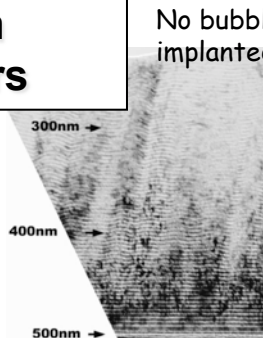
Cu-Nb atomic interface



**He implantation:  
resistance to He  
bubble formation.<sup>1</sup>**

**5 nm  
layers**

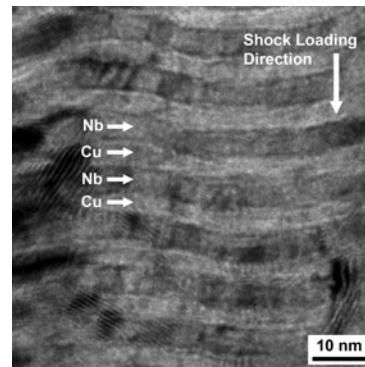
No  
amorphization or  
defect clusters  
after  $10^{17}/\text{cm}^2$   
150keV He  
implantation



No bubbles in He-  
implanted Cu-Nb

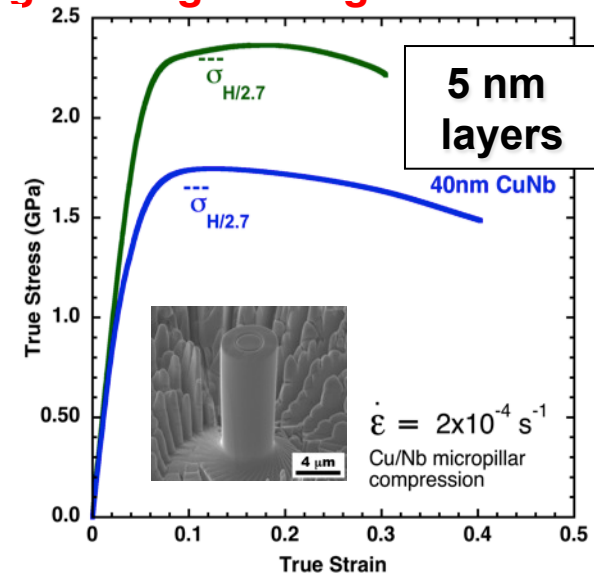
He  
solubility:  
~8%at.

**Laser Shock resistance**

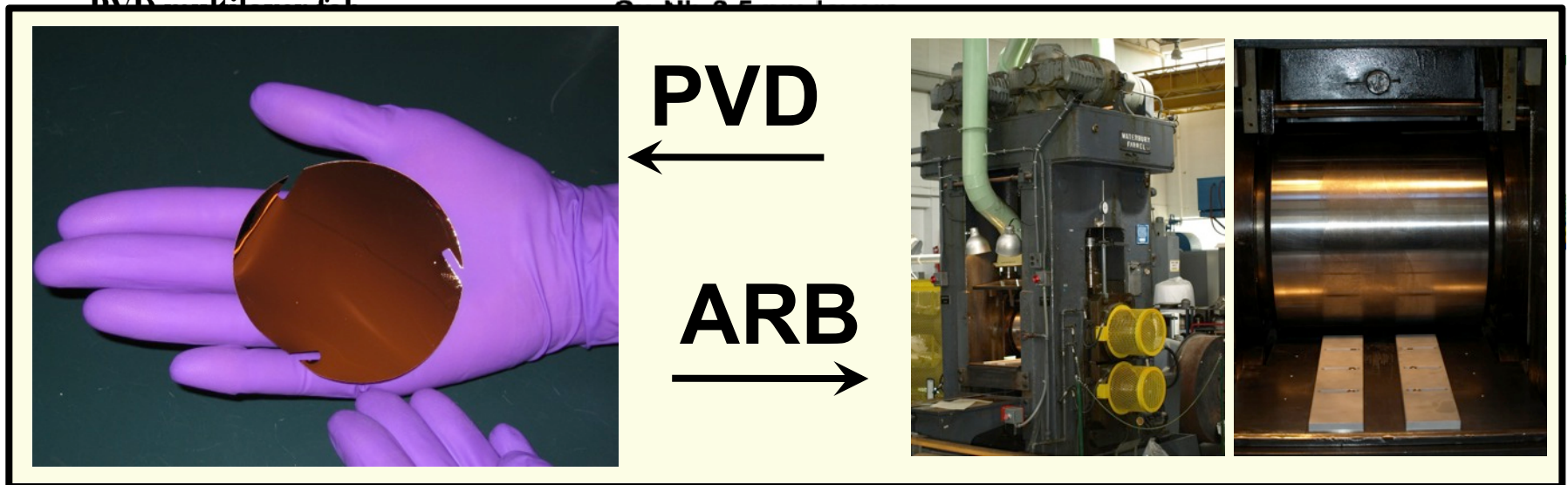


**5 nm layers**

**Micropillar Compression:  
High strength and good ductility<sup>2</sup>**

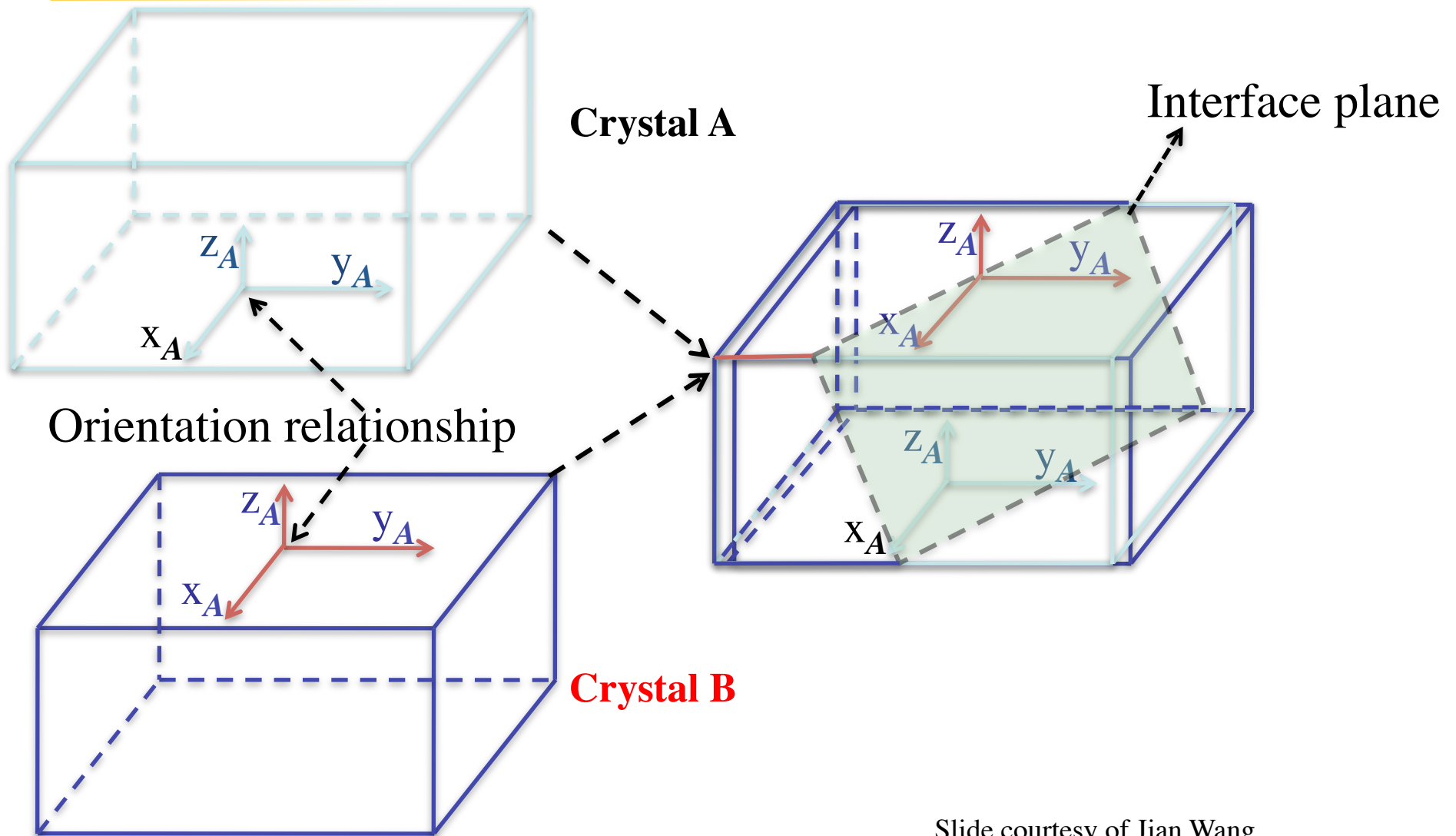


## Extraordinary behavior of Physical Vapor Deposited (PVD) multilayers



**Hypothesis:** The atomic structure of an interface dictates its mechanical response. Interfaces with different atomic structure will exhibit different mechanical behavior. Controlling synthesis routes can give preferred interfacial structures with superior properties.

# Orientation relationship and interface plane

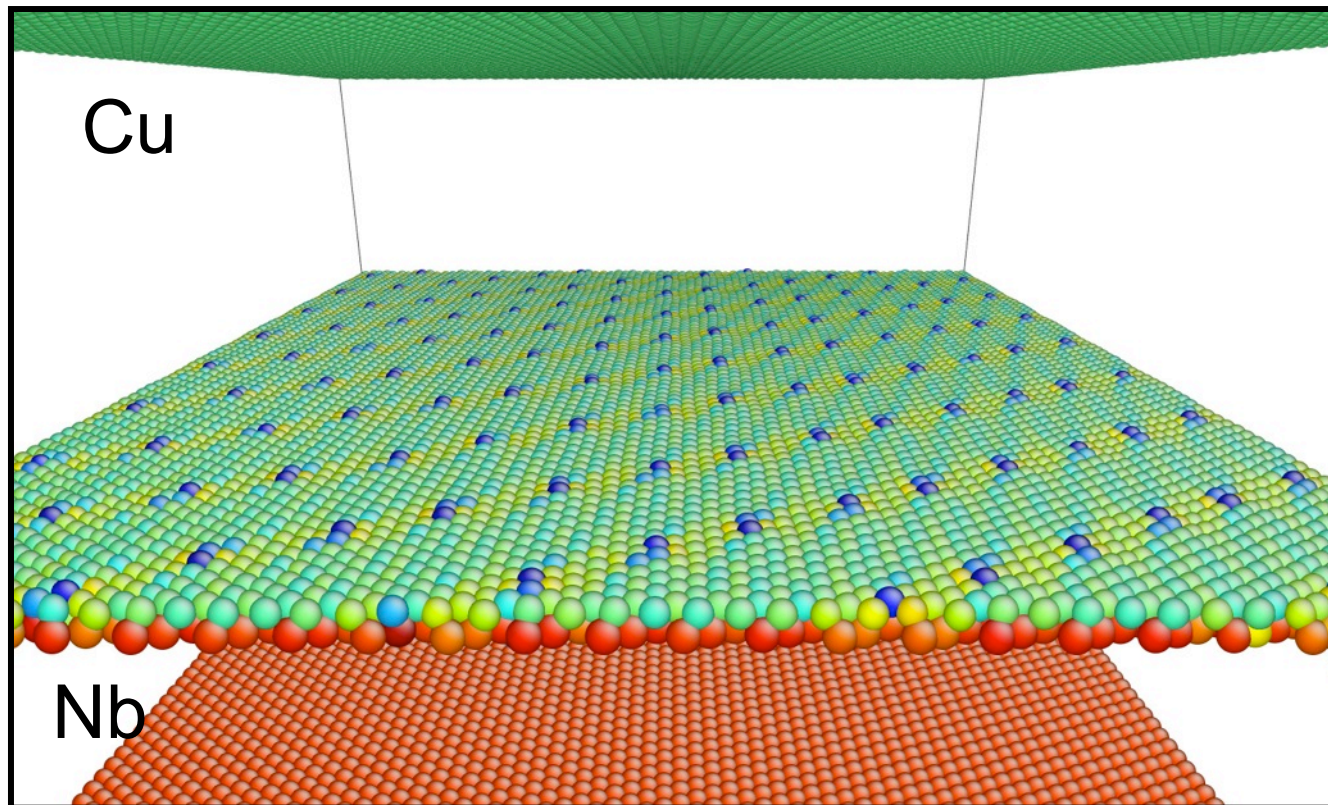


Slide courtesy of Jian Wang



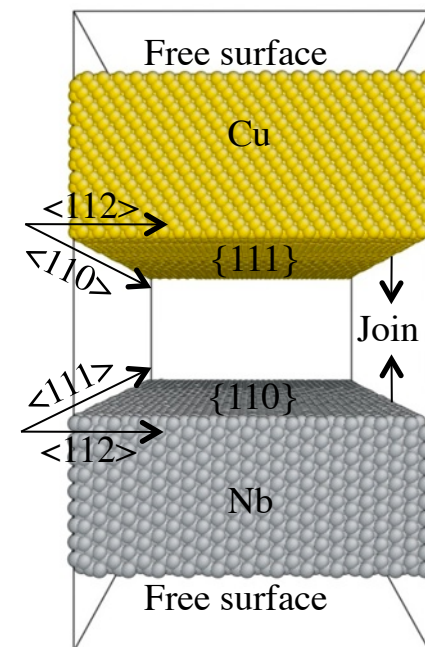
# PVD foils: Fine sample dimensions and relatively uniform interfacial properties

Cu-Nb 2.5 nm layers



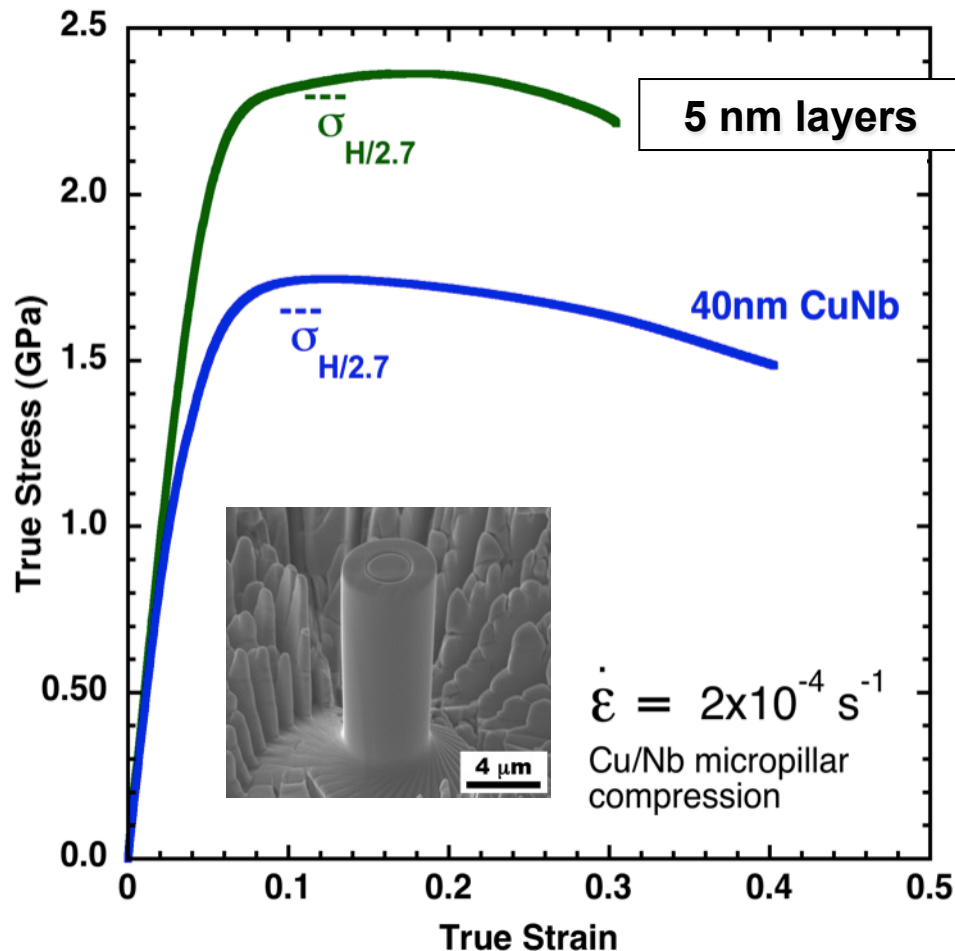
**Characteristic interface in PVD foils**

Interface planes:  
 $\{111\}_{\text{Cu}} \parallel \{110\}_{\text{Nb}}$



## Extraordinary behavior of Physical Vapor Deposited (PVD) multilayers

### Micropillar Compression: High strength and good ductility

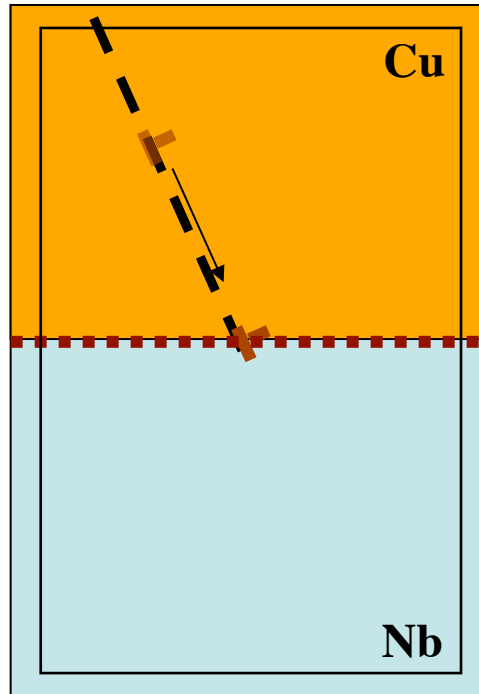


One distinguishing feature makes this material different from pure Cu or Nb

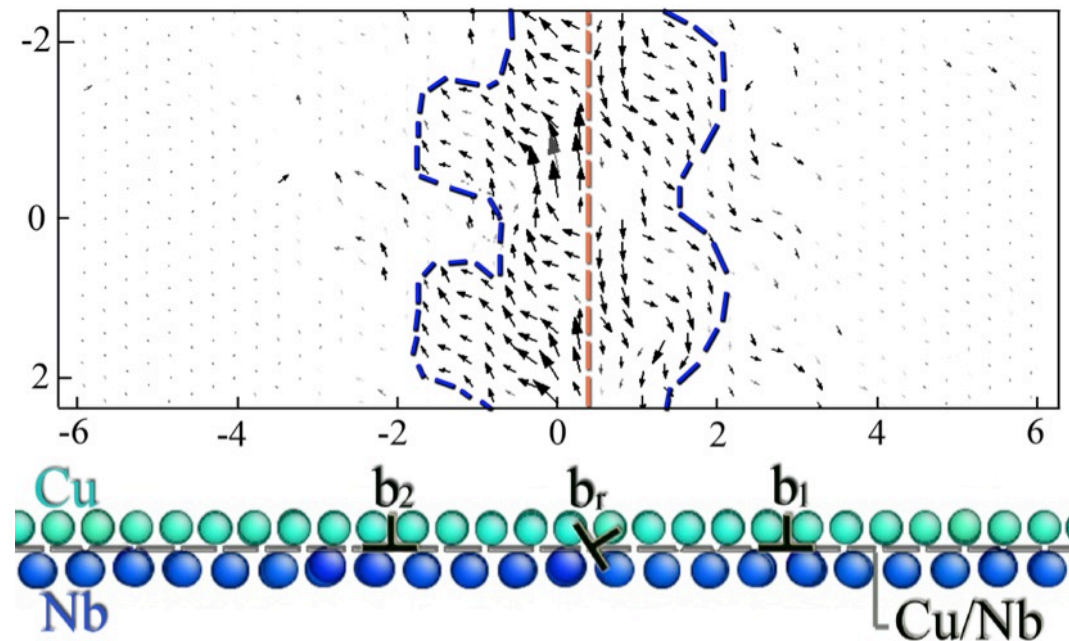
**High interfacial content**

N.A. Mara et al., APL, **92** (2008) 213901.  
N.A. Mara et al., APL, **97** (2010) 021909.

# Glide dislocations are trapped by weak interfaces that shear in response to dislocation stress field



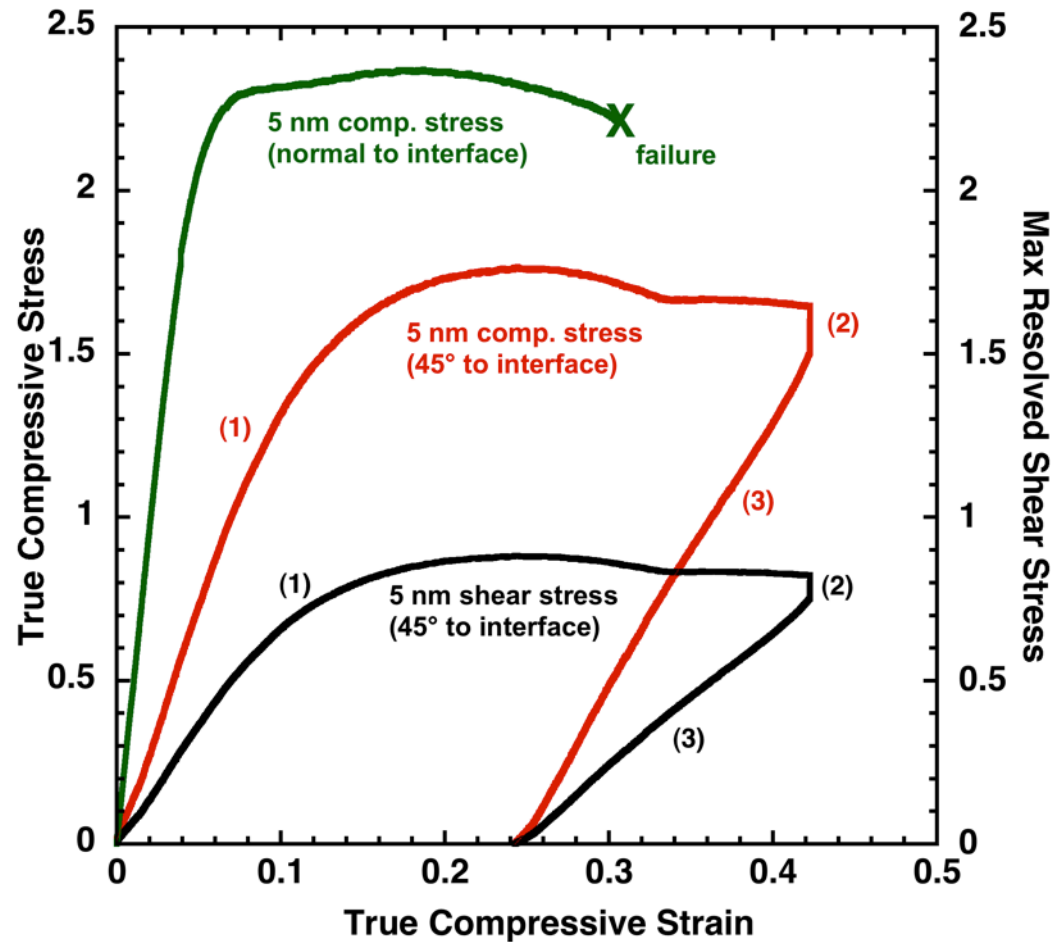
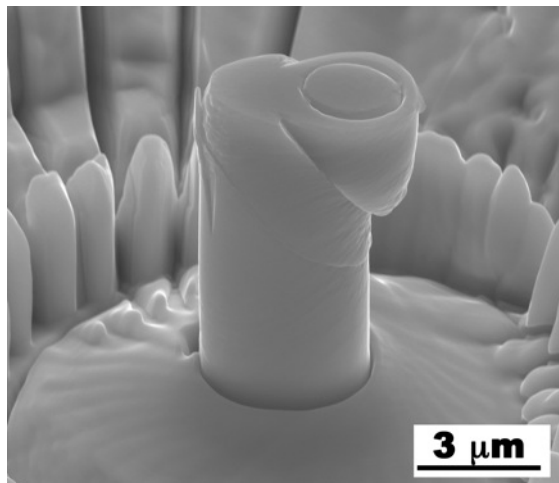
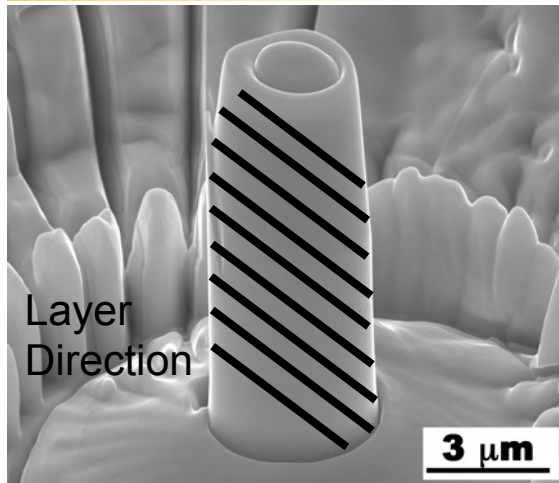
## Core spreading of an edge Shockley partial dislocation within interface



**Dashed lines** indicate the position while the glide dislocation entering interface.  
**Blue dashed curves** outline the region of core spreading within interface.

J. Wang, R.G. Hoagland, J.P. Hirth and A. Misra, Acta Materialia (2008).

## 5 nm CuNb multilayers: layer interface oriented 45° w/r to compression axis





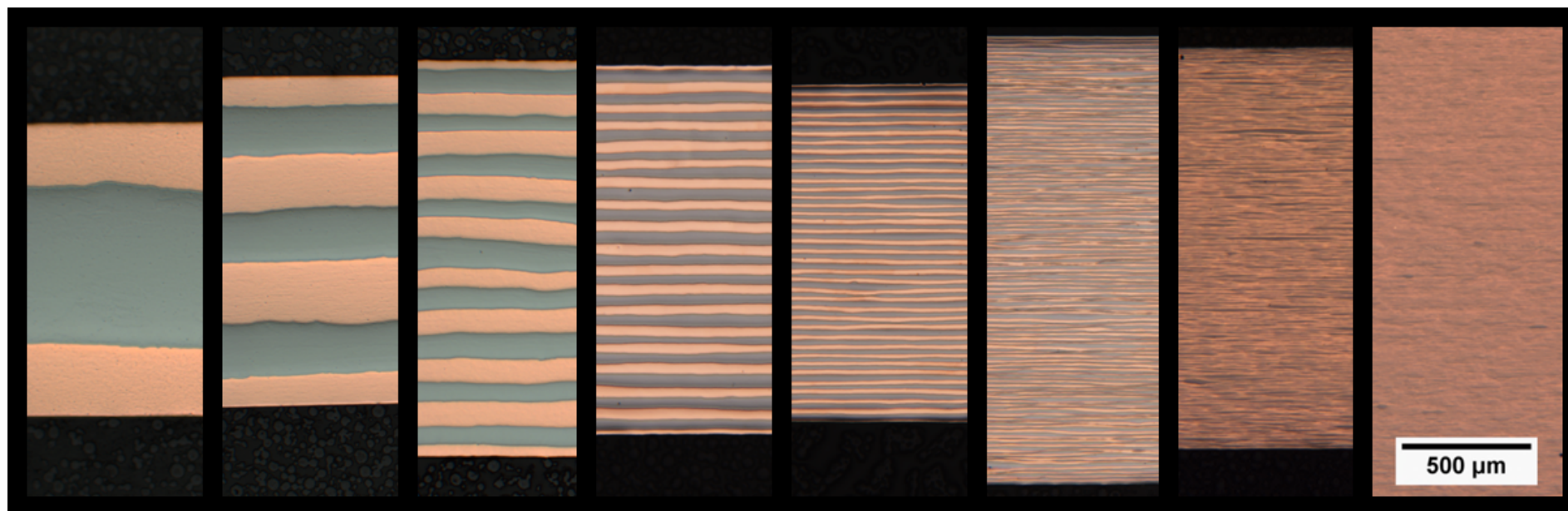
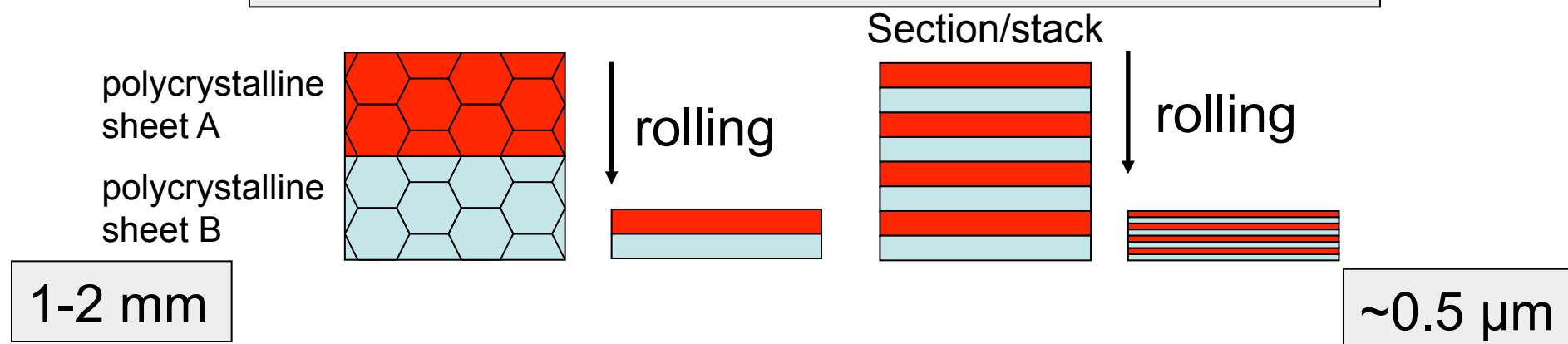
## Physical Vapor Deposited Cu/Nb--Conclusions

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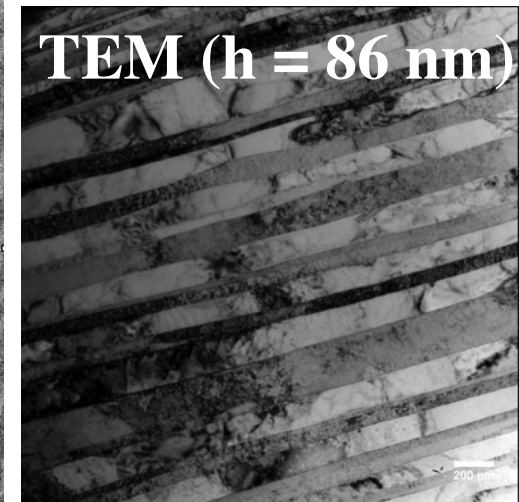
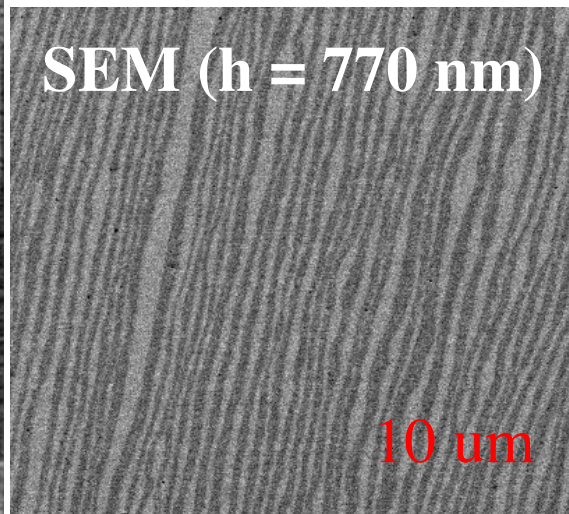
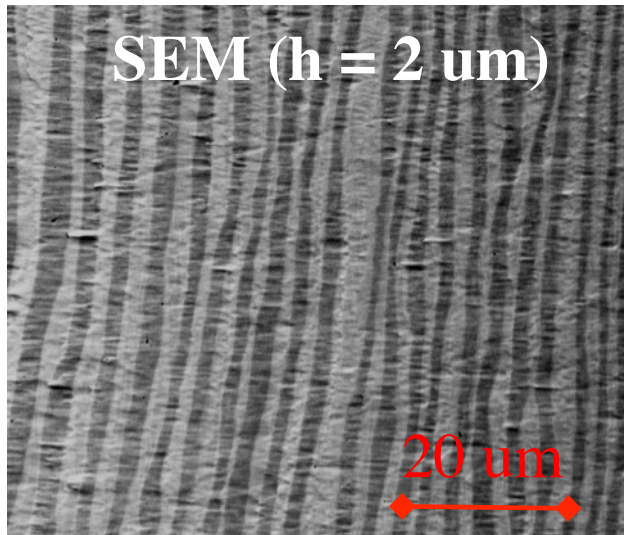
- Atomic structure of the PVD Kurdjumov-Sachs  $\{111\}\text{Cu} // \{110\}\text{Nb}$  interface dictates its mechanical behavior
  - Interface is relatively weak in shear
  - Dislocation core spreading in the interface due to low shear strength makes transmission difficult
  - No deformation twinning evident, even after large strains
- Questions:
  - How will interfaces with differing atomic structure behave under mechanical loading?
  - Effects of:
    - Crystal geometry
    - Misfit dislocation structure
    - Synthesis pathway

# Synthesis of bulk multi-layered nano composite materials chemically identical to PVD

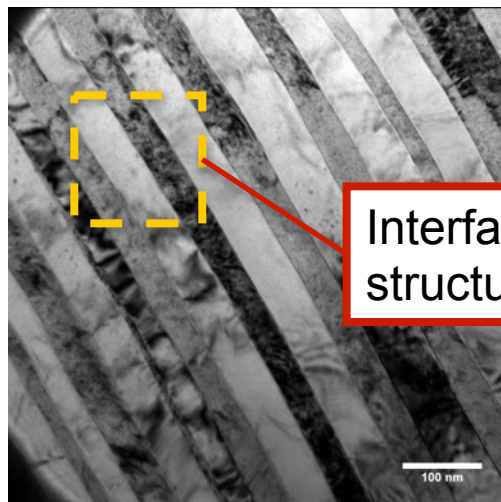
## Accumulative Roll Bonding (ARB): Sheet



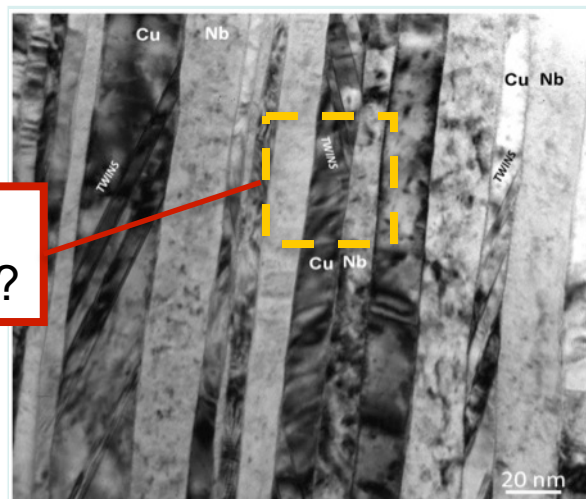
# ARB Cu/Nb composites with controllable layer thickness



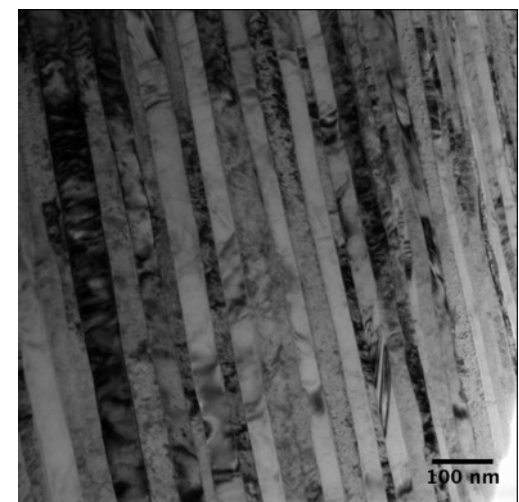
TEM ( $h = 48 \text{ nm}$ )



TEM ( $h = 18 \text{ nm}$ )



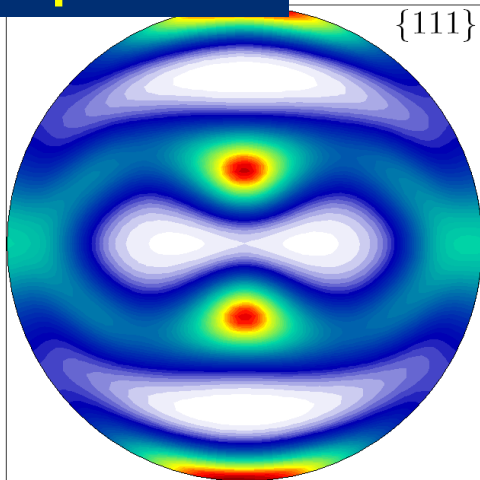
TEM ( $h = 9 \text{ nm}$ )





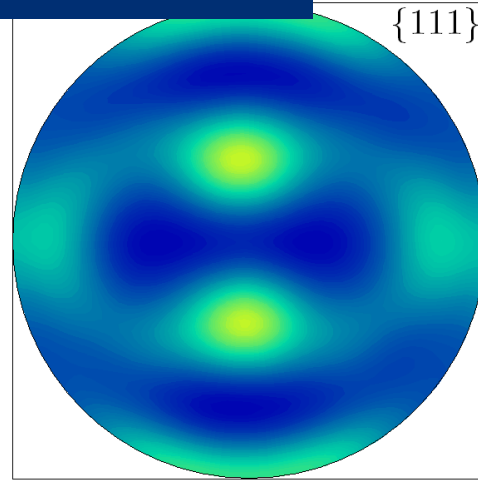
# Deviations from theoretical rolling texture as the layer thickness reduces to nanoscale

**Expected**



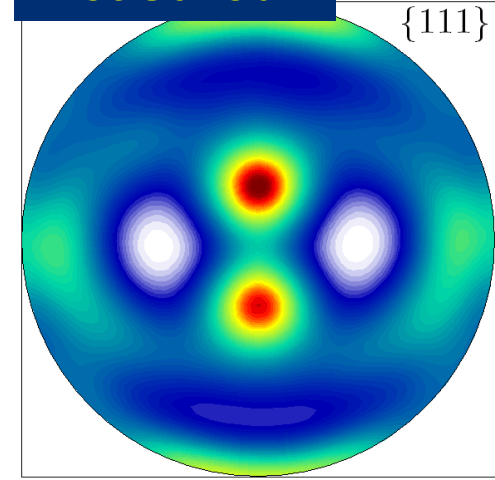
**Cu Rolling Texture**

**Measured**

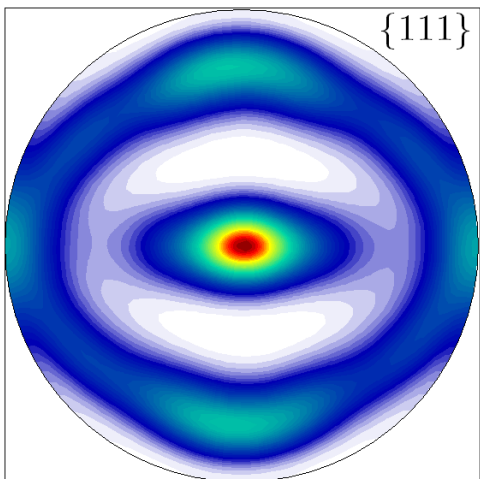


**Cu 2 um As Rolled**

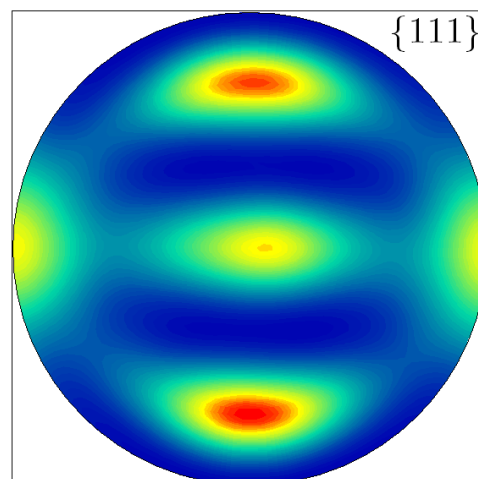
**Measured**



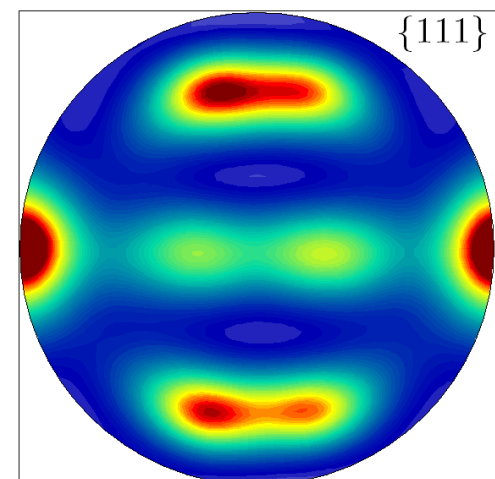
**Cu 18 nm As-Rolled**



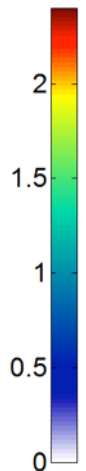
**Nb Rolling Texture**



**Nb 2 um As Rolled**

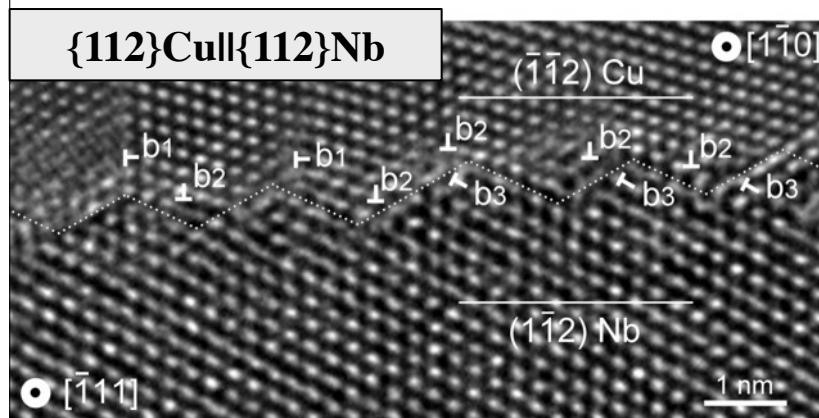


**Nb 18 nm As-Rolled**

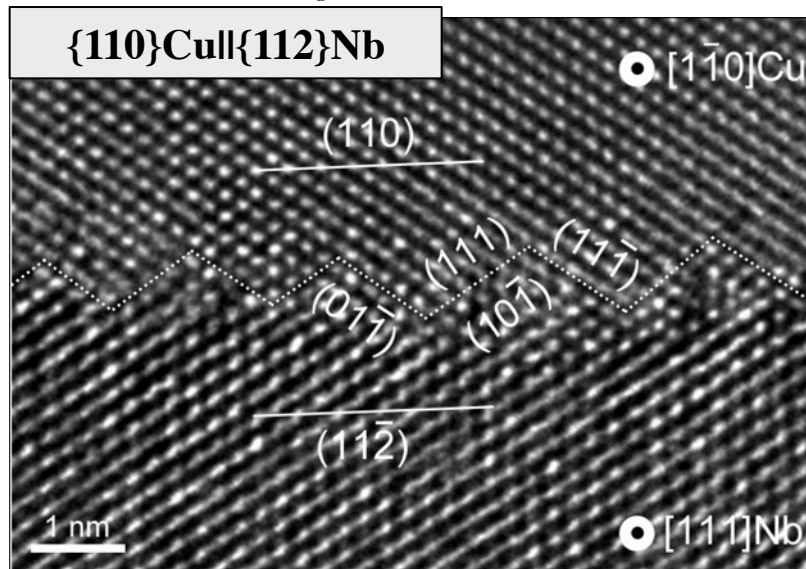




# Two interfaces with same KS orientation relationship but different interface planes



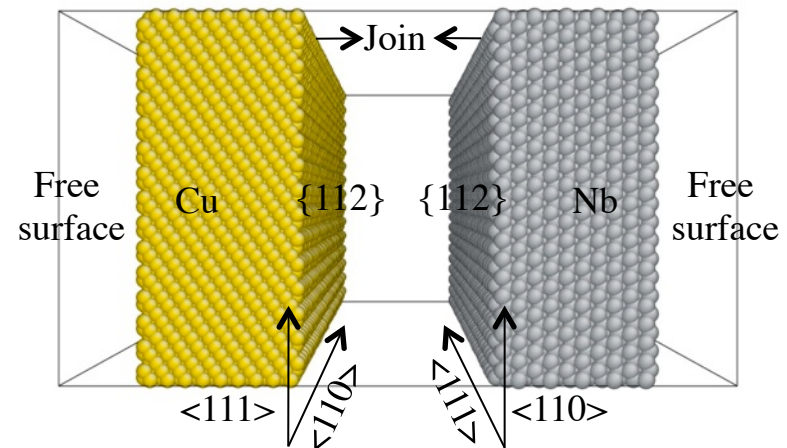
*S. J. Zheng et al. 2012, Acta Mater., In Press*

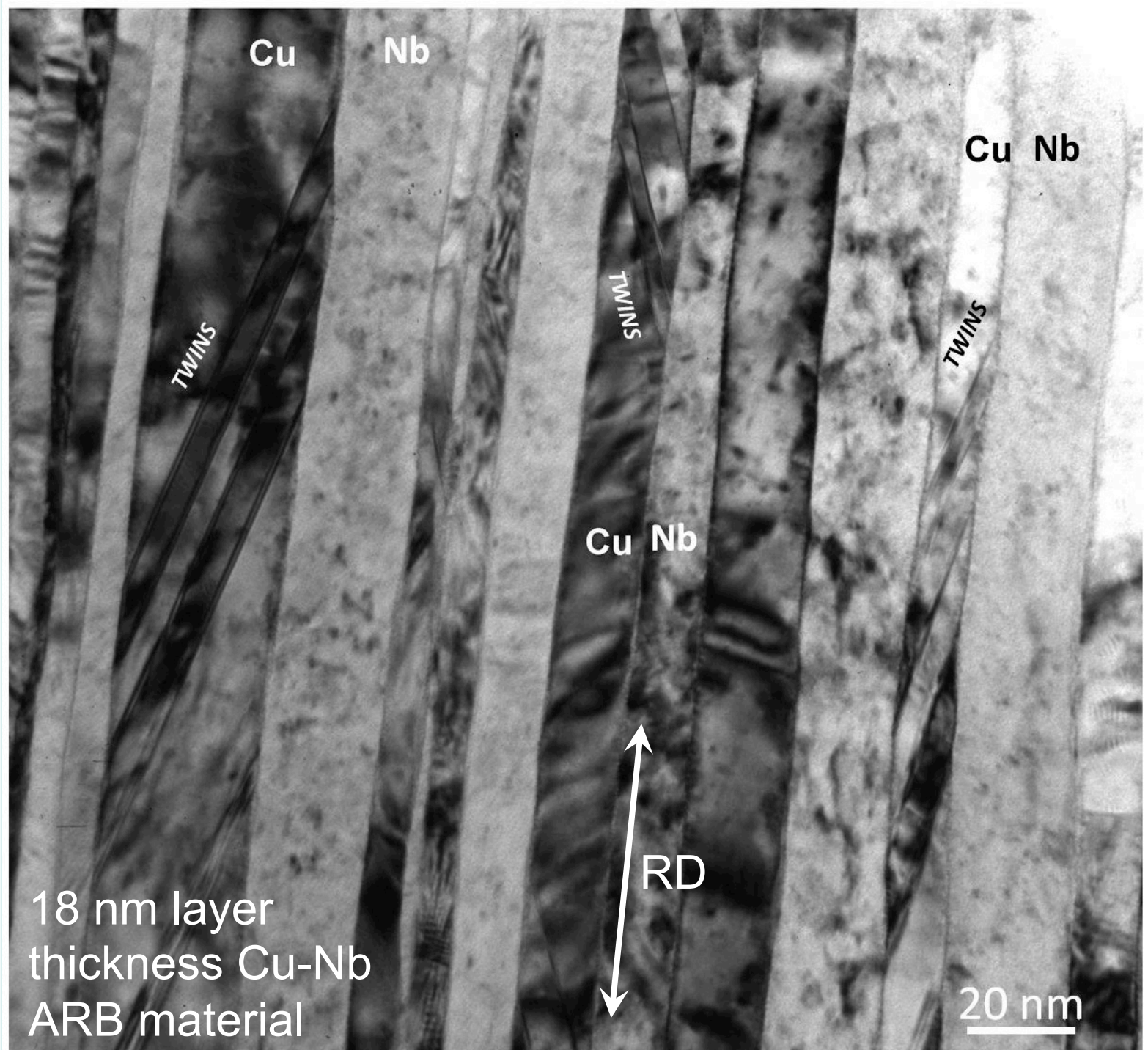


**TD|| <110>Cu||<111>Nb**

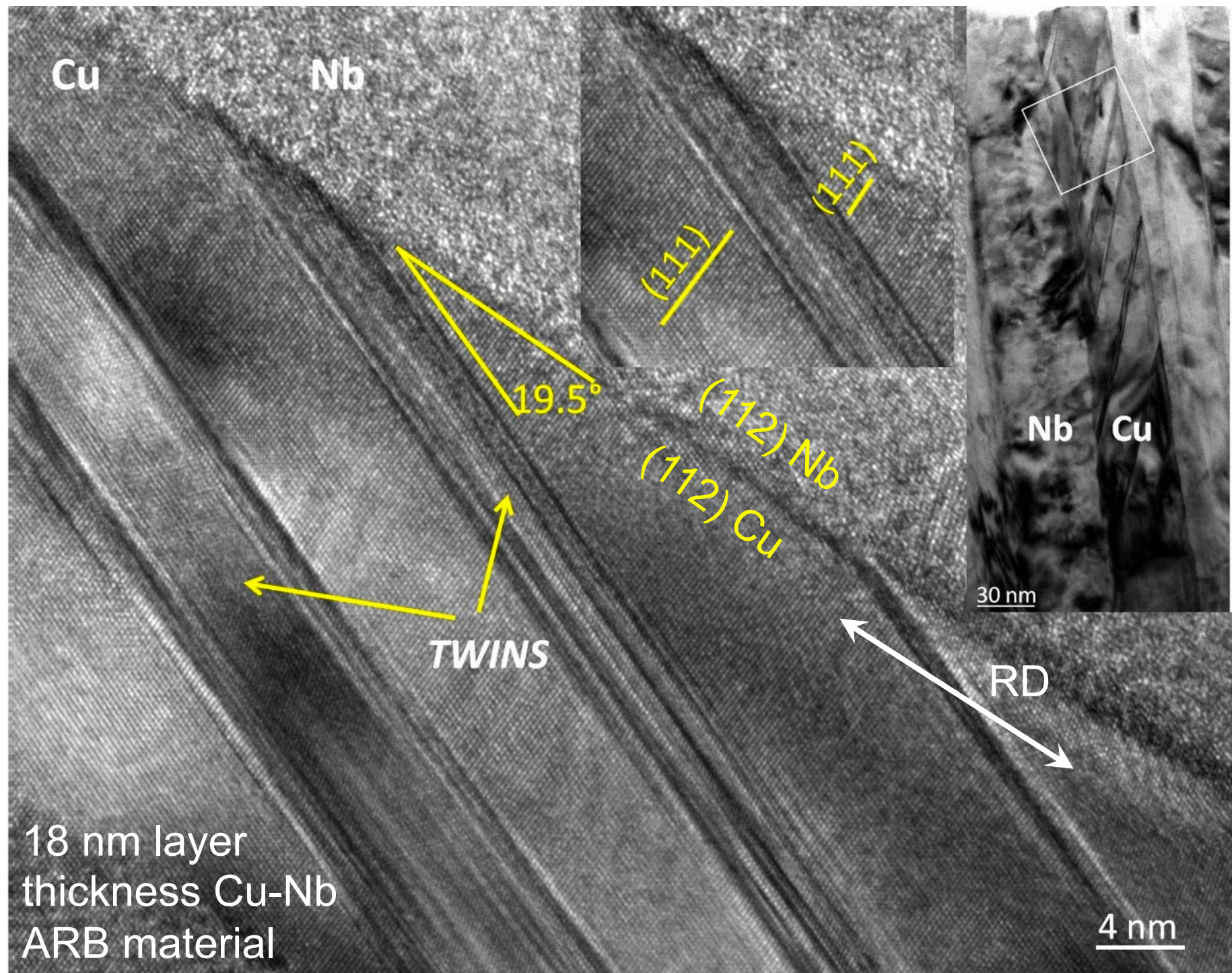
**Characteristic interface in fine ARB samples**

Interface planes:  
 $\{112\}_{\text{Cu}} \parallel \{112\}_{\text{Nb}}$



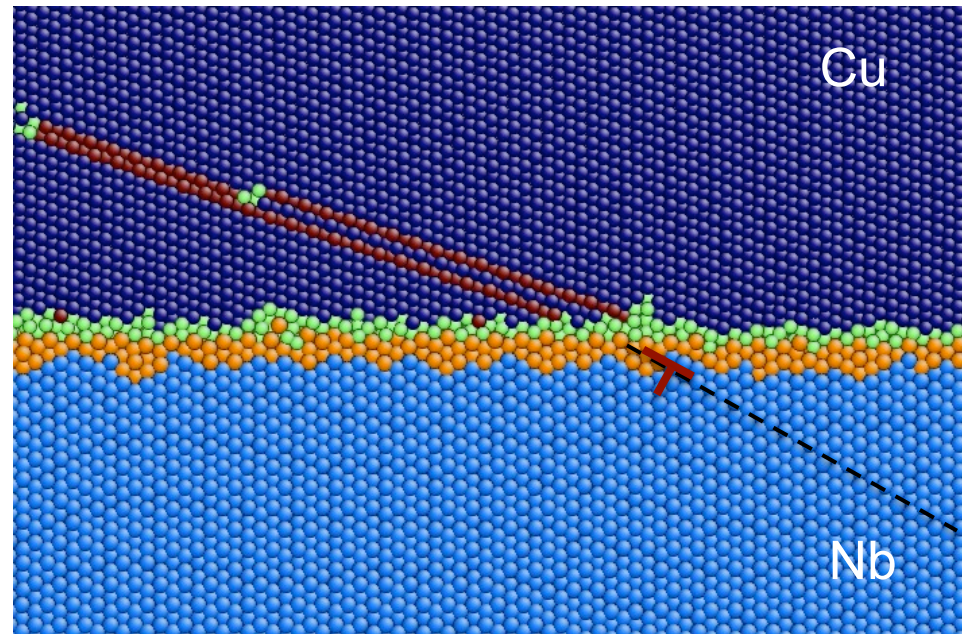
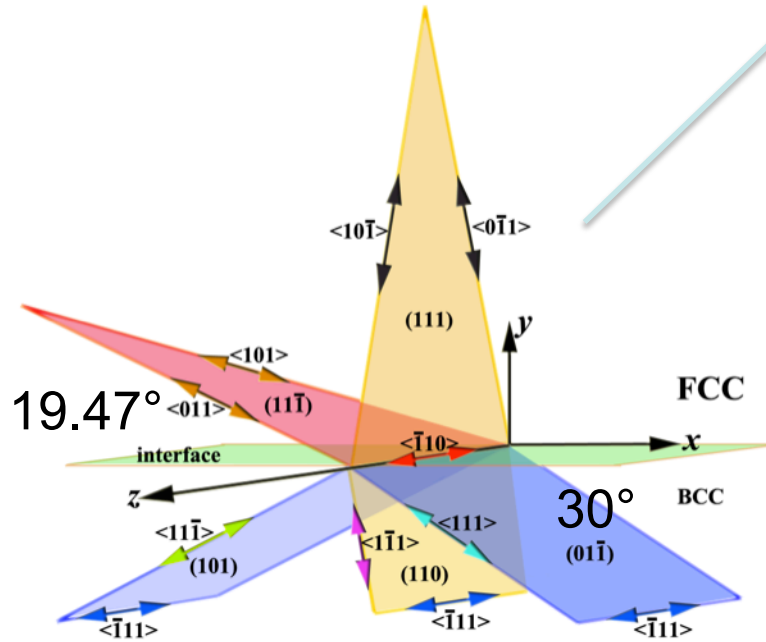
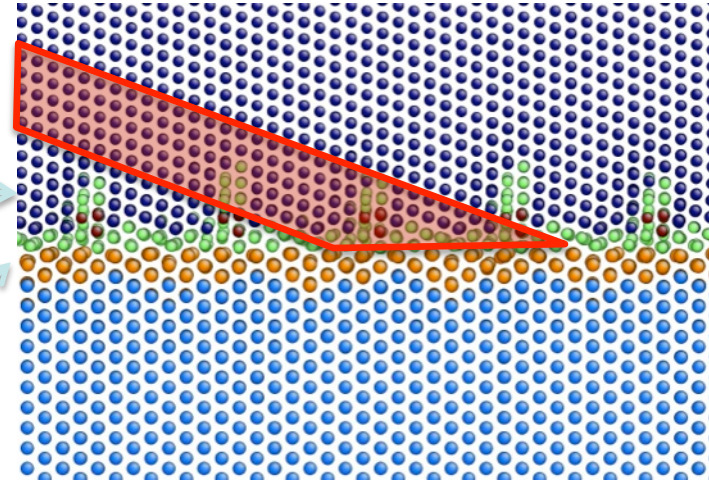
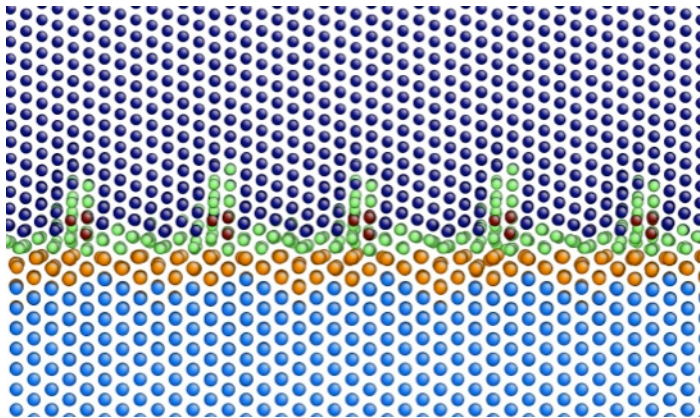








## $\{112\}\text{Cu}/\{112\}\text{Nb}$ KS: Twinning is preferred on the (11-1) slip plane

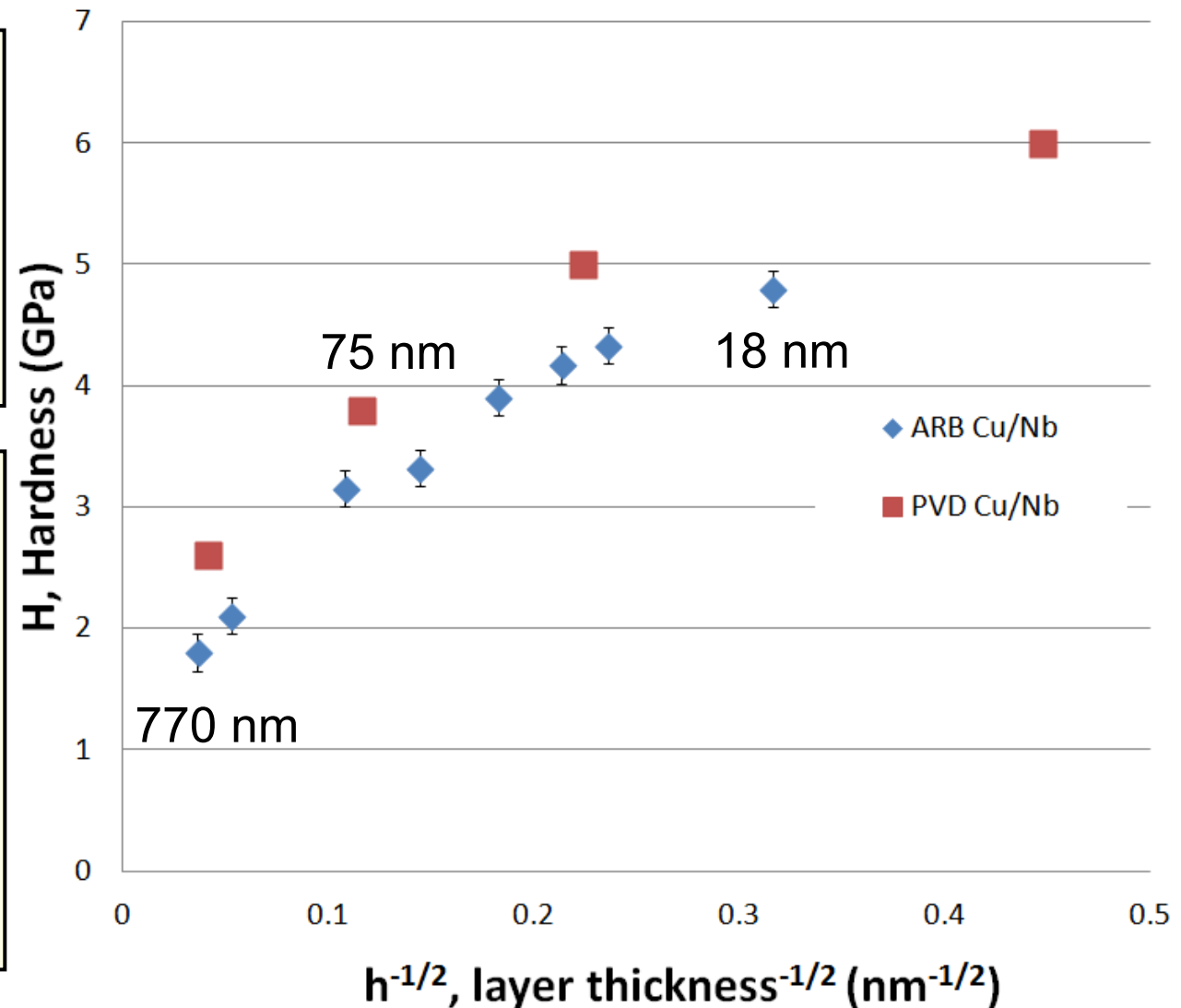




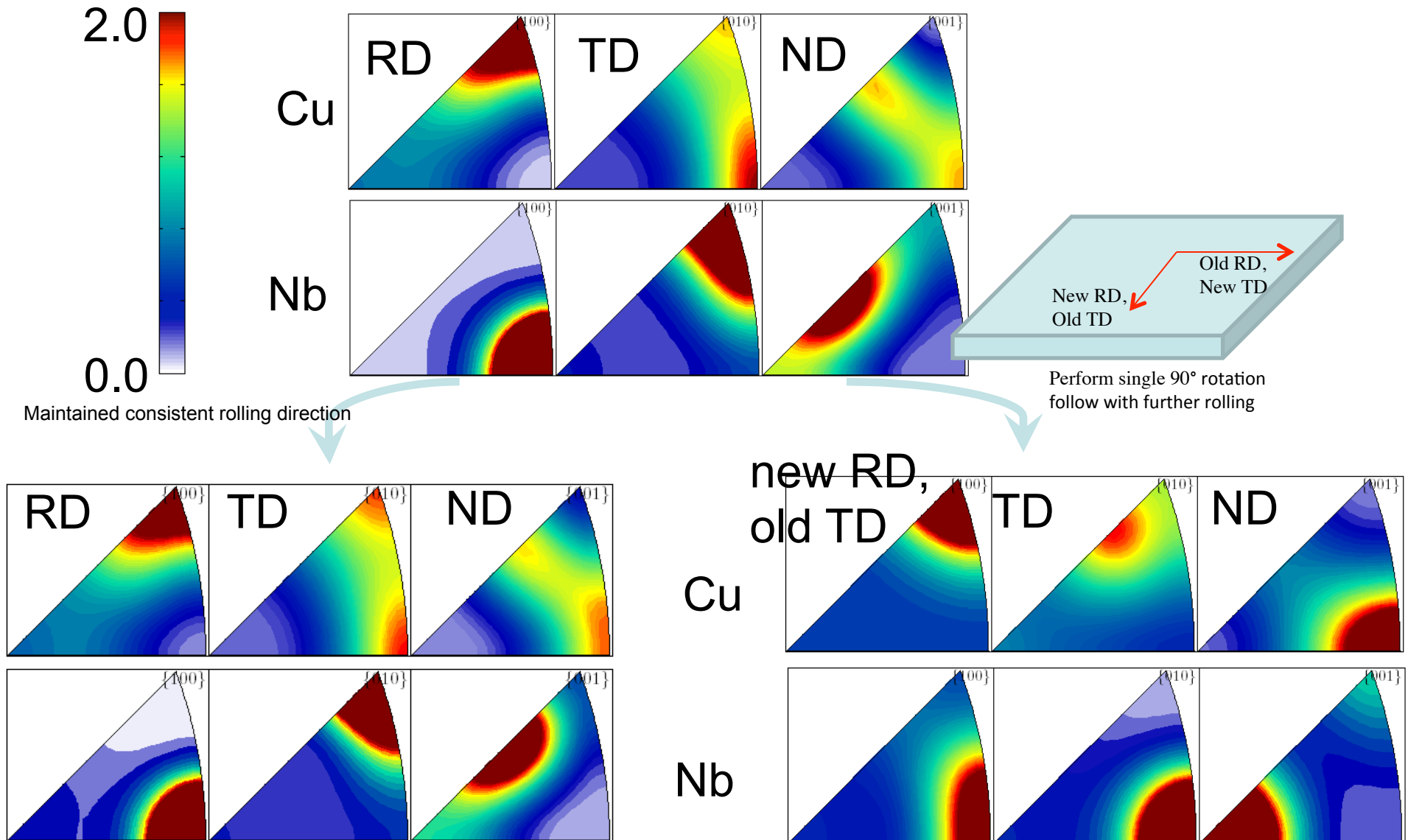
# ARB Material Presents Lower Strength as compared to PVD material

Is the ARB interface a less effective barrier to slip than the PVD interface?

Is the ARB material, with its texture produced via rolling, simply softer in certain directions due to this texture?

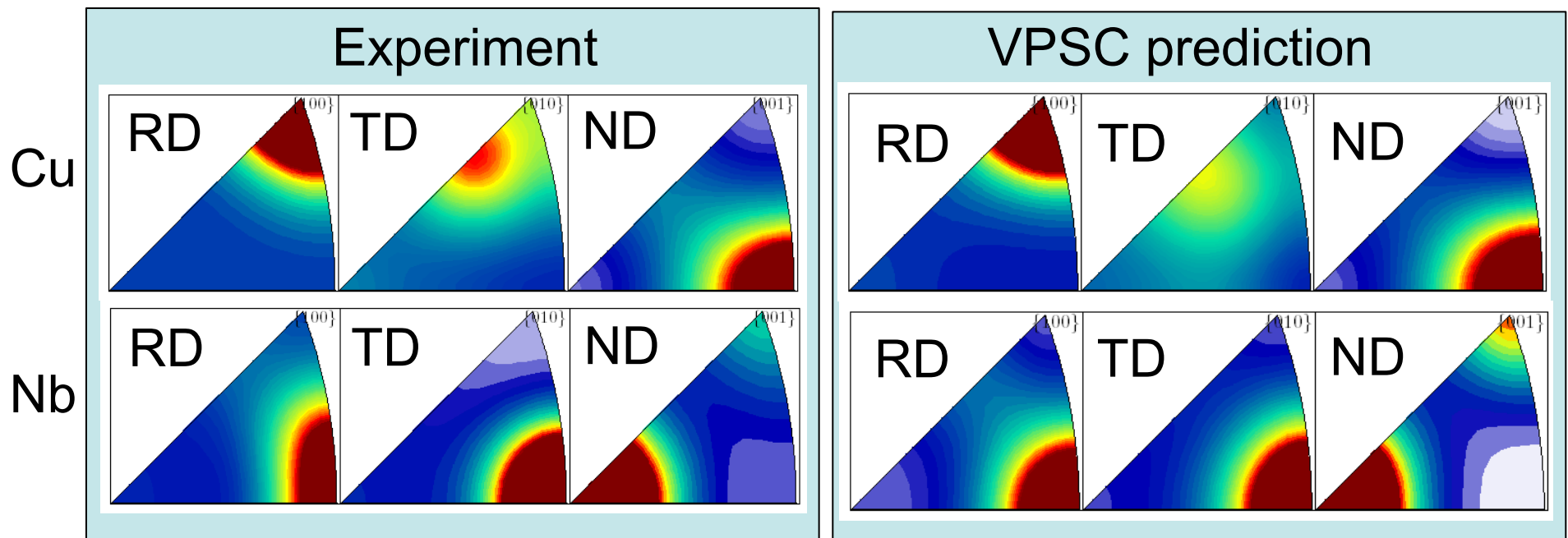


## 90° Strain path change: 56 nm to 30 nm

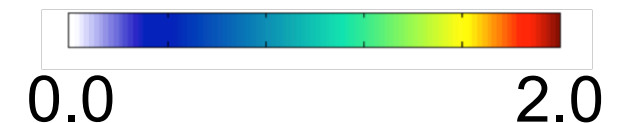


## 90° Strain path change: 56 nm to 30 nm

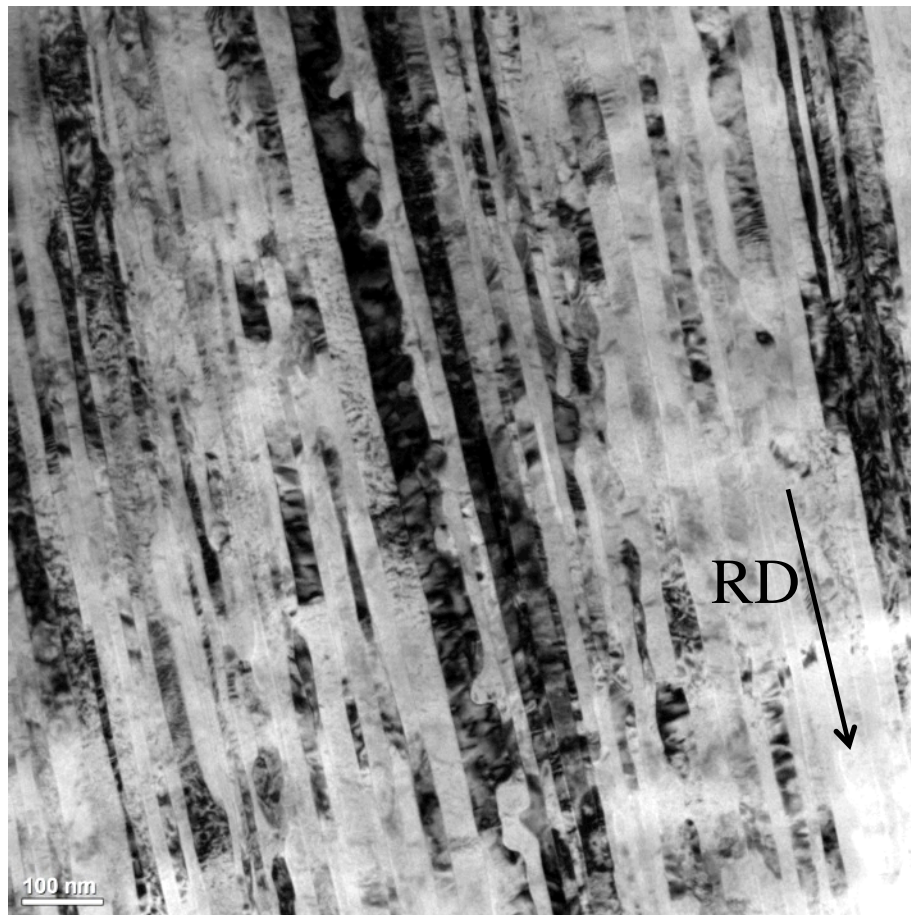
### Inverse Pole Figures after Strain Path Change



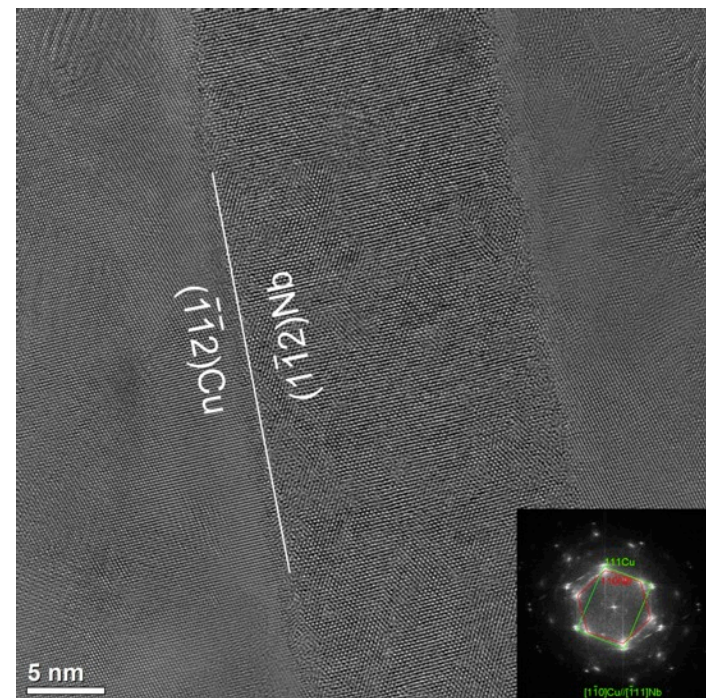
VPSC can predict textures, and can aid in predicting new interface OR/IP



## 18 nm Cu/Nb Annealed at 500°C

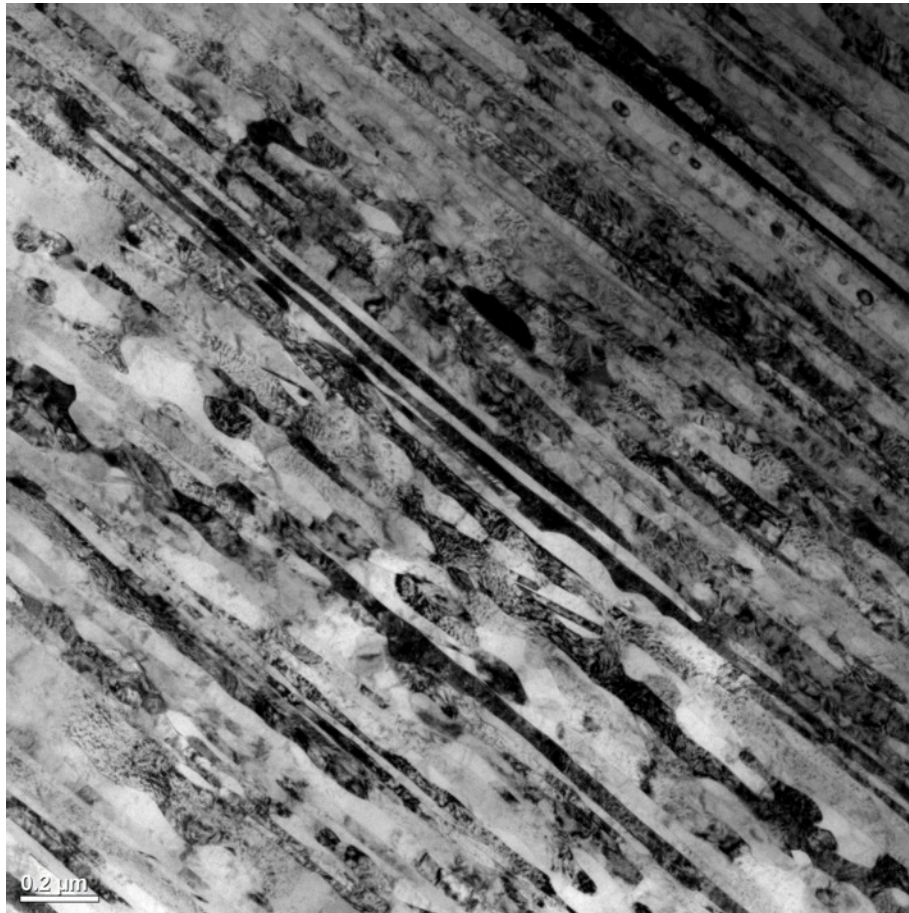


- Layers maintain ~ 18 nm layer thickness
- Interfaces are still largely planar
- Locations do show initial stages of instability, but not at  $(112) \parallel (112)$  interfaces

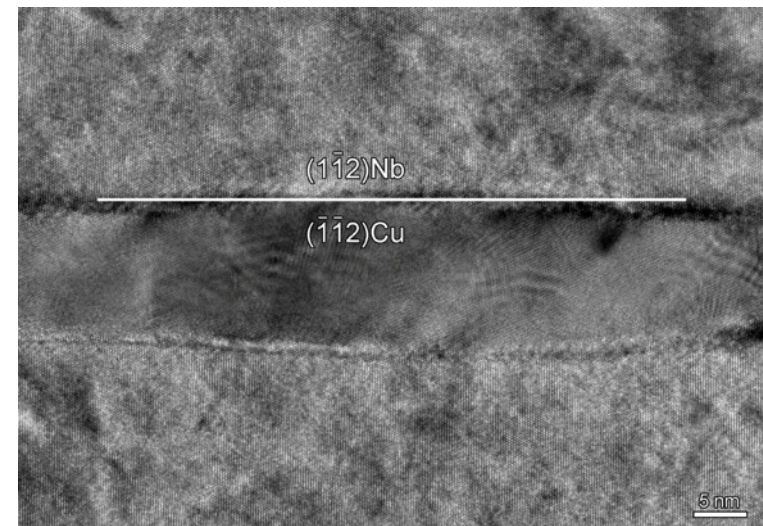




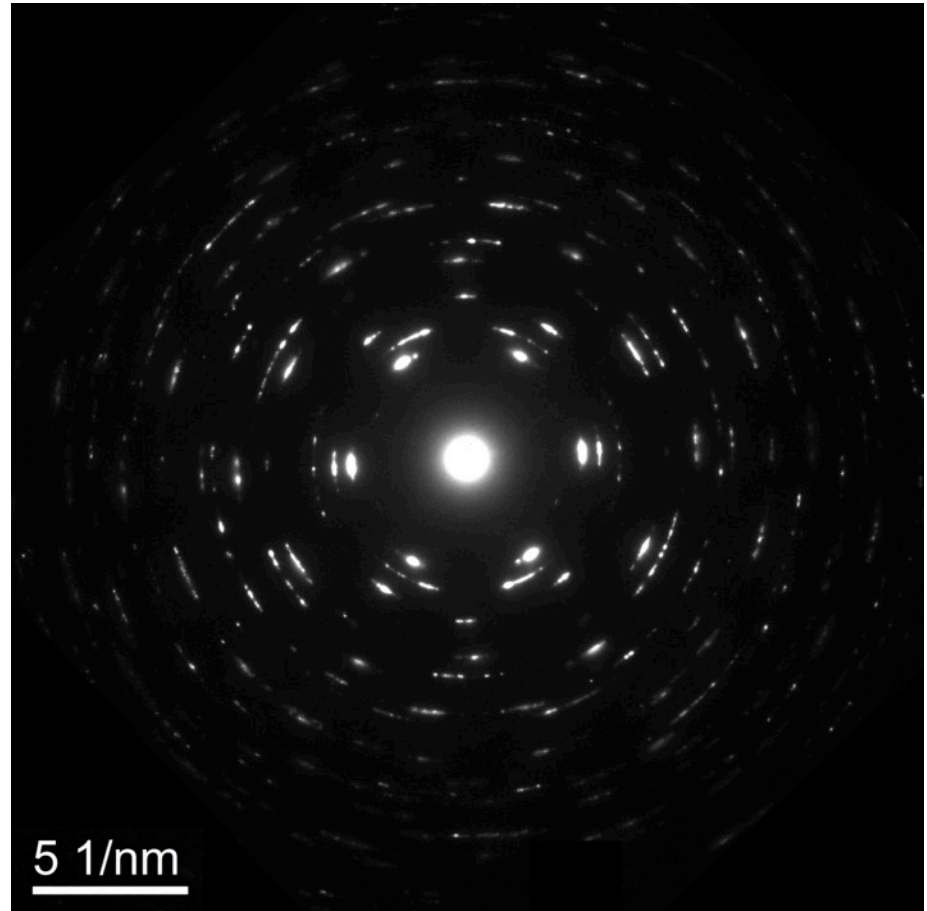
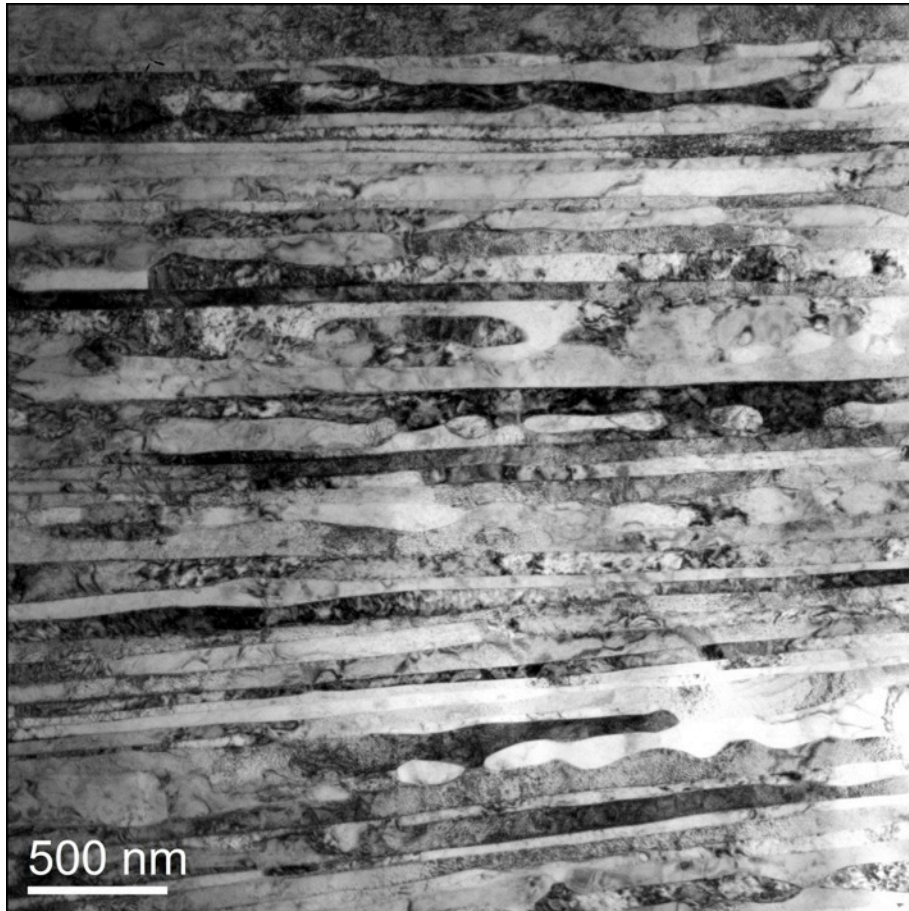
## 18 nm Cu/Nb Annealed at 600°C



- Layers are becoming noticeably thicker ~ 50 nm in thickness
- Interfaces are still largely planar
- More instability noted, but not at  $(112) \parallel (\bar{1}\bar{1}2)$  interfaces



## 18 nm Cu/Nb Annealed at 700°C



Strong texture and lamellar structure is maintained

Figure 1 is a scatter plot with error bars showing the relationship between Hardness (H, GPa) and the inverse of the square root of the layer thickness ( $1/h^{1/2}$ , nm $^{-1/2}$ ). The y-axis ranges from 2 to 5 GPa, and the x-axis ranges from 0 to 0.25 nm $^{-1/2}$ . Three data series are plotted: 900C (350 nm) in green, 700C (70 nm) in red, and 48 nm in blue. Each data point includes a vertical error bar. Three orange boxes above the data points indicate the layer thicknesses: 364 nm for 900C, 86 nm for 700C, and 48 nm for 48 nm. Two insets show TEM images of the nanolaminates. The left inset shows a low-magnification view of the 900C laminate with a 0.5  $\mu$ m scale bar. The right inset shows a high-magnification view of the 48 nm laminate with a 200 nm scale bar and an arrow indicating the rolling direction.

Sample	Layer Thickness (nm)	$1/h^{1/2}$ (nm $^{-1/2}$ )	Hardness (H, GPa)
900C	350	~0.05	~2.7
700C	70	~0.12	~3.3
48 nm	48	~0.14	~3.3

Lamellar structure is important in maintaining trend in hardness

## Conclusions

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- As-processed ARB material has similar morphology, chemistry as PVD, but different interfacial structure
- Density of interfaces AND interfacial structure play a role in determining hardness
  - Example: twinning in Cu at the  $\{112\}\text{Cu} // \{112\}\text{Nb}$  interface
  - Higher strength, no twinning in Cu in the  $\{111\}\text{Cu} // \{110\}\text{Nb}$  interface
- Need to understand effects of processing history to predict the effects on the interfaces we produce:
  - Amount of strain
  - Strain Path
  - Annealing